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TENTATIVE REFERENCE METHOD FOR THE MEASUREMENT OF GROSS ALPHA AND GROSS BETA RADIOACTIVITIES IN ENVIRONMENTAL WATERS

National Environmental Research Center
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16. ABSTRACT

A tentative reference method for the measurement of gross alpha and gross beta radioactivities in environmental waters is described. Samples of environmental water sources are collected, preserved by acid treatment, and aliquots of the samples are evaporated to dryness in a counting dish and counted for alpha and beta activity. Counting efficiencies for sample aliquots are read from curves prepared from counting data of prepared standards, using a known quantity of cesium-137 and 0-10 milligrams of evaporated water dissolved solids per square centimeter of counting dish area for gross beta, and a known quantity of americium-241 and 0-5 milligrams of evaporated water dissolved solids per square centimeter of counting dish area for gross alpha. Results are reported in pCi/liter.

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TENTATIVE REFERENCE METHOD FOR THE MEASUREMENT OF GROSS ALPHA AND GROSS BETA RADIOACTIVITIES IN ENVIRONMENTAL WATERS

1. Principle and Applicability

Measurable amounts of alpha and beta emitting radionuclides are found in most environmental waters. These may be naturally occurring radionuclides such as uranium, radium, thorium, and potassium-40, or they may be the result of man-made (artificial) radionuclides. These naturally occurring elements (radium and thorium), through their daughter gases, cause an appreciable airborne particulate activity which may contribute to the radioactivity of environmental waters through fallout (carried by rain or snow). Artificial radionuclides may be released to the environment through fallout or by contamination of process and waste water (resulting) from nuclear reactors, weapons testing, medical waste, and nuclear reprocessing plants.

Just as there are differences in the toxicities of the various stable elements, there are differences in the radiotoxicities of the various radionuclides. Radium-226 and strontium-90 are two radioisotopes, found in potable water, with very high radiotoxicities.

The 1962 Public Health Service Drinking Water Standards recommended limits of 3 pCi/liter for radium-226 and 10 pCi/liter for strontium-90. In these standards an upper limit of 1000 pCi/liter of gross beta activity (in the absence* of alpha emitters and strontium-90) was set. However, the drinking water guidelines currently under consideration by the EPA set limits for radium-226 and radium-228 at 5 pCi/liter and

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* Absence is intended to mean a negligible small fraction of the limits established for these radionuclides and the limit for unidentified alpha emitters is taken as the listed limit for radium-226.

gross alpha activity (including radium-226) at 15 pCi/liter. Gross beta limit is set at 50 pCi/liter corrected for potassium-40 and other naturally occurring radionuclides.

Methods for the analysis of radium-226 and strontium-90 are complex and time consuming. There are large numbers of water sources that must be checked routinely for radioactivity. Therefore, the measurement of gross alpha and gross beta radioactivities provides a rapid screening method which will indicate or negate the need for specific analysis of the more hazardous radionuclides (radium-226 and strontium-90).

This method describes the measurement of gross alpha and gross beta radioactivities and provides for the analysis of radionuclides in both the soluble and insoluble fractions of environmental water samples. Radionuclides that are volatile under the sample preparation conditions required by this method will not be measured.

2. Range and Sensitivity

The method is applicable to the measurement of alpha emitters having energies above 3.9 megaelectronvolts (MeV) and beta emitters having maximum energies above 0.1 MeV.

The minimum limit of concentration to which this method is applicable will depend on sample size, counting system characteristics, background, and counting time.

The decay of radioactivity is random in nature rather than uniform. Therefore, the emissions of radioactive decay (alpha and beta particles, and gamma photons) must be counted sufficiently long to obtain the desired statistical reliability. It is recommended that samples be counted long enough so that samples with activities as low as the detection limit of the method and counting instrument used will have a counting error of plus or minus (±) counts per minute (cpm) at the 95 percent confidence level of no more than the sample net count rate

(when counting error cpm equals net cpm, the counting error is 100 percent). For instance, when a 1-liter water sample aliquot containing 200 milligrams (mg) of sample residue solids is evaporated in a 2-inch diameter counting dish and counted for 20 minutes for gross beta activity in an external proportional counting system having a background of 2 cpm, with a counting efficiency of 30 percent, and the gross count rate is 3 cpm, the sample will have a net count rate of 1 cpm and a counting error of ± 1 cpm at the 95 percent confidence level. This corresponds to a detection limit of 1.5 ± 1.5 pCi/liter. Also, when a 1-liter water sample aliquot containing 87 mg of sample residue solids is evaporated in a 2-inch diameter counting dish and counted for 50 minutes for gross alpha activity in an alpha scintillation system having a background of 0.005 cpm, with a counting efficiency of 30 percent, and the gross count rate is 0.09 cpm, the sample will have a net count rate of 0.085 cpm and a counting error of ± 0.085 cpm at the 95 percent confidence level. This corresponds to a detection limit of 0.13 ± 0.13 pCi/liter. Counting efficiency will increase with less sample residue resulting in a correspondingly lower detection limit. Samples having activities above the detection limits and counted for the times indicated above (20 minutes for gross beta and 50 minutes for gross alpha) will have correspondingly lower counting error. See the Appendix.

3. Interferences

Since the radionuclides are not separated from the evaporated sample residue, the sample residue serves as an interference, absorbing alpha and beta particles emitted from the radionuclides. Moisture adsorbed or trapped by the sample residue also is an interference for the reason stated above. If a sample is counted in an internal proportional counter, static charge on the sample residue can cause erratic counting, thereby preventing an accurate count.

Non-uniformity of the sample residue in the counting dish interferes with the precision of the method.

4. Precision and Accuracy

4.1 Gross alpha and gross beta measurement by this method has a built-in inaccuracy in that samples may contain alpha and beta emitters with energies different from the standards used. In such circumstances the counting efficiencies used will not give accurate information (counts per minute to disintegrations per minute) for the radionuclides in the sample. However, the method at best is good only for screening or checking trends. The importance of precision (or repeatability) is that a given water source will be checked periodically for gross alpha and beta and any significant changes in the results from one time to the next may require specific analysis for radium-226, strontium-90, or other suspected radionuclides. It is necessary then that such changes be real and not a result of poor precision.

Water samples containing no more than 200 mg of evaporated (dried) residue per liter with gross beta activity as low as 50 pCi/liter can be analyzed with a precision of less than ± 10 percent at the 95 percent confidence level (200 mg of sample residue in a 2-inch diameter counting dish, counted for 20 minutes in a thin-window external proportional gas flow counting system, with a counting efficiency of 30 percent).

Water samples containing no more than 87 mg of evaporated (dried) residue per liter with gross alpha activity of 5 pCi/liter can be analyzed with a precision of less than ± 10 percent at the 95 percent confidence level and with gross alpha activity of 500 pCi/liter, a precision of less than ± 6 percent at the 95 percent confidence level (87 mg of sample residue in a 2-inch diameter counting dish, counted for 50 minutes in a thin-window external proportional gas flow counting system, with a counting efficiency of 6 percent).

Precision of the gross alpha analysis can be improved by counting the samples in an alpha scintillation system (mounting a zinc sulfide

film directly on the sample residue, held in close contact by an o-ring in the counting dish, and counting in a light-tight system with a photomultiplier tube detector).

Analytical results of spiked water samples prepared to contain as low as 50 pCi/liter of cesium-137 (for gross beta) and 5 pCi/liter of americium-241 (for gross alpha), analyzed by this method, and counted in an external thin-window proportional counting system, should show accuracies with deviations from the known values of less than ± 10 percent at the 95 percent confidence level.

4.2 Counting Instruments

4.2.1 Internal Proportional Counter

There are inherent problems with this type of counting system; however, it has been widely used in the past. Some problems in this type of system are poor repeatability in counting due to static charges building up on the sample surface and detector contamination from loose sample residue. Also, it does not lend itself to automatic changing of samples. A thin film of Lucite is dried on the sample residue to hold the residue in place. Then a conductive film is also dried on the Lucite film to reduce the buildup of static charges (1). Both coatings add solids to the sample, thus adding to the sample self-absorption of the emitted alpha and beta particles. Conductive solutions presently available do not seem to improve substantially the counting repeatability.

4.2.2 External Proportional Counter, Thin Window

This type of counting system is much the same as the internal proportional counting system except that a thin window separates the sample from the counting chamber. The window then acts as an absorber; however, the window can be made sufficiently thin to minimize absorption. There is also air space between the sample dish and the detector

window which absorbs to some extent, especially alpha particles. However, this type of counting system does give good repeatability in counting and is, therefore, a recommended type of counting instrument. Instruments of this type are available that have low background counts and automatic sample changing.

4.2.3 Scintillation Counter

The detector for this type of counting system consists of a photomultiplier tube on which is mounted a zinc sulfide (silver activated) coated film for alpha detection or a thin (0.020-inch) plastic phosphor disc for beta detection. For alpha counting, efficiency can be increased by placing the zinc sulfide film on or very close to the sample instead of on the phototube. The cost of the zinc sulfide film is low enough to allow for a one-sample use (about 20 cents for a 1-15/16-inch diameter film which can be used with a 2-inch diameter counting dish). The cost of plastic phosphor discs is too high to allow for a one-sample use (about $13.50 per disc).

This type of counting system does not lend itself to automatic sample counting as well as the external proportional type because the photomultiplier tube must be protected from light whenever high voltage is applied. The scintillation-type counting system does give good counting repeatability, good counting efficiencies, and has low background. Therefore, this is a recommended type of counting system. Such systems for both alpha and beta scintillation counting have been described in the literature (2,3).

5. Apparatus

One-liter (or one-quart) tight-sealing polyethylene sample bottles

Tags or labels for sample identification

Filter holder for 47-millimeter filters

47-millimeter diameter 0.45 micrometer membrane filters (cellulose triacetate type)

Two-liter filter flask with stopper adapter for filter holder

Tygon tubing (3/8-inch ID x 5/8-inch OD)

Graduated cylinders, 100, 250, and 1000 milliliter

Suction pump

Four-ounce polyethylene bottles with Poly-Seal caps. To be used for taking portions of 8N nitric acid into the field for sample collection (15 milliliter of 8N nitric acid per liter of sample)

Stainless steel counting dishes (2-inch diameter by 1/4-inch deep) and o-rings to fit tight inside of dishes

Drying oven

Desiccator and desiccant

Alpha and beta counting systems, such as:

- Internal proportional counter - windowless
- External proportional counter - thin window
- Scintillation counting system:
  - Phototube with zinc sulfide film detector for alpha counting
  - Phototube with thin plastic scintillator disc for beta counting

Beakers, 250 and 400 milliliter

Zinc sulfide film discs, 1-15/16-inch diameter (available from William B. Johnson and Associates, Inc., Research Park, Montville, New Jersey 07045)

Plastic scintillator disc, 2-inch diameter (available from Nuclear Enterprises, 935 Terminal Way, San Carlos, California 94070)

6. Reagents. All chemicals should be of "reagent-grade" or equivalent whenever they are commercially available.*

Concentrated nitric acid (16N)

1+1 nitric acid-water solution (volume dilution) (8N)

1+30 nitric acid-water solution (volume dilution) (0.5N)

30 percent hydrogen peroxide

7. Procedure

7.1 Sampling

Sampling may be accomplished as described in "Environmental Radioactivity Surveillance Guide," published by the U.S. Environmental Protection Agency as report ORP/SID 72-2. It is recommended that samples be preserved at the time of collection by the addition of 15 ml of 1+1 nitric acid-water solution (8N) per liter of sample collected. This will make the sample normality about 0.1N and should reduce loss of sample radioactivity to the container walls. However, if the radioactivity in the separate dissolved and suspended fractions of the sample is to be measured, those fractions must be separated before a preservative is added since a preservative may change the distribution

* "Reagent Chemicals, American Chemical Society Specifications," American Chemical Society (ACS), Washington, DC. For reagents not listed by the ACS see "Reagent Chemicals and Standards" by Joseph Rosin, D. Van Nostrund Company, Inc., New York, NY, or the "United States Pharmacopeia" for purity tests.

of the sample radioactivity. Rather than separating the dissolved and suspended solids fractions in the field, samples can be brought to the laboratory without perservative added. Separation is then done by filtering out the suspended solids on a tared 0.45 micrometer membrane filter. The filter is dried, weighed, and set aside for further analysis. The preservative solution is then added to the filtrate which is returned to the original sample container and held there overnight or longer before analysis is begun.

7.2 Analysis

7.2.1 Sample Size and Counting Efficiency

Sample residue self-absorption must be considered for both alpha and beta radioactivities when choosing the sample aliquot size. Therefore, the analyst must know the mass abundance of the dissolved and the suspended solids fractions of each water sample to be analyzed. Such a determination is made by taking a measured aliquot of the water sample, filtering it through a weighed 0.45 micrometer pore size membrane filter, evaporating the filtrate in a tared beaker, drying the filter and filtrate residue, and reweighing both to get increase due to the dissolved and suspended solids fractions of the water sample. Once the residue fractions have been determined for a given water source there is no need to repeat the determination unless the source has noticeably changed.

For each counting instrument to be used the analyst should prepare a graph of water sample residue versus counting efficiency using known amounts of cesium-137 for beta radioactivity and americium-241 for alpha radioactivity, varying the sample residue from 0 to 5 mg per square centimeter (sq cm) of counting dish area for alpha and 0 to 10 mg per sq cm for beta. See Calibration (Section 8).

7.2.2 Gross Alpha and Gross Beta Measurement of the Combined Solids (Dissolved and Suspended)

7.2.2.1 If gross alpha and gross beta counts are to be made from separate water sample aliquots, use an aliquot size that will result in no more total solids than 10 mg per sq cm of counting dish area for beta and 5 mg of solids per sq cm for alpha. It follows then, that if both alpha and beta measurements are to be made from the same water sample aliquot that the aliquot-solids limitation should be 5 mg per sq cm of counting dish area.

7.2.2.2 Before taking aliquots, shake the sample bottle to mix its contents.

7.2.2.3 To count an aliquot of the water sample, either evaporate to dryness in small increments directly in a tared counting dish or transfer the aliquot to a Pyrex beaker, evaporate to a small volume, quantitatively transfer the concentrate to a tared counting dish with 0.5N nitric acid and then evaporate to dryness. Do not allow the sample residue to go to dryness in the beaker. A uniform deposition of the sample residue in the counting dish is necessary for obtaining reliable comparative counting data. This can be achieved by carefully evaporating the sample concentrate in the counting dish on a variable-temperature hot plate at a sufficiently low temperature to prevent the sample from boiling or spattering. Evaporate the sample concentrate in small portions (not more than 5 milliliters (ml) at a time) to minimize solids deposition on the dish walls.

7.2.2.4 Dry the sample residue in a drying oven at 105° C for at least 2 hours, cool in a desiccator, weigh, and count. If the counting of the sample is to be delayed, store the sample residue in a desiccator until the counting is to be done. Sample residue should be very dry when counting is done because vapors from moist residue will attack the detector window and/or detector.

7.2.2.5 Count for alpha and beta radioactivities at their respective voltage plateaus. If the sample is to be recounted for decay information, store it in a desiccator.

7.2.2.6 For water sources that are turbid, it is recommended that a sample aliquot be wet-ashed to reduce the solids. Evaporate a sample aliquot to near dryness and then treat the residue with one or more portions of 20 ml of concentrated nitric acid plus 2 ml of 30 percent hydrogen peroxide, heating and evaporating to near dryness with each treatment. Then continue with the sample according to steps 7.2.2.3 through 7.2.2.5.

7.2.3 Gross Alpha and Gross Beta Measurements of the Separate Dissolved and Suspended Solids

7.2.3.1 These samples should be filtered or preservative should be added to them before they are sent to the laboratory. The dissolved and the suspended solids fractions are separated by filtering the sample through a weighed 0.45 micrometer pore size membrane filter. Return the dissolved solids fraction (filtrate) to the original container, add 15 ml of 8N nitric acid per liter of sample, and wait overnight or longer before continuing the analysis on the dissolved solids fraction. Place the filter in a weighed counting dish and place a weighed o-ring in the dish to hold the filter flat (o-ring should be tight fitting in the dish). Dry the filter for 2 hours at 105° C, cool to room temperature and reweigh. An aliquot of the dissolved solids is treated as in Section 7.2.2. If the suspended fraction weighs not more than 5 mg per sq cm of counting dish area, count as is for gross alpha and gross beta, and normalize the count to the aliquot size used for the dissolved solids fraction. If the suspended fraction weighs more than 5 mg per sq cm of counting dish area, that fraction plus the filter should be wet ashed by the following step.

7.2.3.2 Wet ashing of suspended solids can be accomplished by one or more treatments with 20 ml of concentrated nitric acid and 2 ml of 30% hydrogen peroxide in a 250-ml beaker, heating and evaporating to near dryness with each treatment. Transfer the wet-ashed residue with 0.5N nitric acid to a tared counting dish and continue with drying and counting as in Section 7.2.2. If the wet-ashed residue is still more than 5 mg per sq cm of counting dish area, then redissolve the residue, dilute to a known volume, and take an aliquot equal to or less than 5 mg per sq cm of counting dish area and proceed as in Section 7.2.2.

8. Calibration

Using standard solutions of cesium-137 (for beta radioactivity), americium-241 (for alpha activity) and tap water, prepare reference data for counting efficiency versus water solids per unit area of counting surface. Add known amounts of alpha and beta radioactivity to varying size aliquots of tap water. Evaporate the water aliquots, transfer the solids concentrate to tared counting dishes, dry, and count according to the procedure of Section 7.2.2. Correct the counting data for background activity and plot counting efficiency versus mg of solids per sq cm of counting dish area. Such a curve should be determined for each counting instrument for both alpha and beta and should cover the range of 0-10 mg per sq cm for beta and 0-5 mg per sq cm for alpha. Counting efficiencies for calculating gross alpha and gross beta radioactivities of environmental water sources are read from these curves.

9. Calculations and Reporting

It is recommended that the gross alpha and gross beta radioactivities be determined and reported in pCi/liter. When counting beta radioactivity by gas flow proportional counting systems, alpha radioactivity present in the sample (or sample fraction) is counted

also unless the particular instrument being used is designed and has been set for discriminating out the alpha pulses (higher energy). Without instrumental discrimination it is necessary to subtract the alpha activity (which had been counted at the alpha voltage plateau) from the beta plus alpha activity (counted at the beta voltage plateau). Determine alpha radioactivity by the following equation:

$$\text{Alpha (pCi/liter)} = \frac{\text{net cpm} \times c_1}{c_2 e V}$$

where net cpm = gross alpha counts minus the background counts at the alpha voltage plateau

c_1 = 1000 ml/liter

c_2 = 2.22 dpm/pCi

e = efficiency fraction, read from graph of efficiency versus mg of water solids per sq cm of counting dish area (alpha curve)

V = volume of the aliquot analyzed, in ml

Determine beta radioactivity by the following equation:

$$\text{Beta (pCi/liter)} = \frac{\text{net cpm} \times c_1}{c_2 e V} - \text{alpha (pCi/liter)}^*$$

where c_1 = 1000 ml/liter

c_2 = 2.22 dpm/pCi

e = efficiency fraction, read from graph of efficiency versus mg of water solids per sq cm of counting dish area (beta graph)

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* The subtraction of alpha activity is necessary if the beta count is performed without instrumental discrimination for alpha counts.

net cpm = gross counts at the beta plateau voltage minus the background counts at the beta plateau voltage

Total water sample gross alpha and gross beta radioactivity is the sum of dissolved solids and suspended solids radioactivities if separated and analyzed separately.

Error associated with the results of the analysis should also be reported. See the appendix for error and statistical calculations.

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APPENDIX. ERROR AND STATISTICAL CALCULATIONS

Because of the random nature of radioactivity disintegrations there is an error associated with any measured count of these disintegrations. The variability of any measurement is indicated by the standard deviation. The standard deviation in the counting rate is determined by the following equation:

$$\sigma\,(R) = \left(\frac{R_0}{t_1} - \frac{B}{t_2}\right)^{\frac{1}{2}}$$

where R_0 = gross count rate

t_1 = counting time for the gross count

B = background count rate

t_2 = counting time for the background count

The counting error for a given sample expressed in pCi/liter and at the 95% confidence level is shown by:

$$E = \frac{c_1\sigma(R) \times c_2}{c_3 eV}$$

where c_1 = 1.96 = 95% confidence factor

c_2 = 1000 ml/liter

c_3 = 2.22 dpm/pCi

e = efficiency factor, cpm/dpm

V = volume of the aliquot analyzed, in ml

The standard deviation associated with the overall random uncertainty may be estimated by performing replicate analyses on a given sample, and is calculated from the following equation:

$$S_n = \left[\sum_{i=1}^{m} (n_i - \bar{n})^2/(m - 1)\right]^{\frac{1}{2}}$$

where n = activity (pCi/liter) of a given sample

$\bar{n}$ = mean activity (pCi/liter) of a series of analyses

m = the number of replicate analyses